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SOURCE SAMPLING: METHODS AND EQUIPMENT

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SOURCE SAMPLING: METHODS AND EQUIPMENT

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The primary problem in particulate sampling is to avoid selective sampling of particular sizes due to inertial effects. This will become clear when examining the diagrams shown.

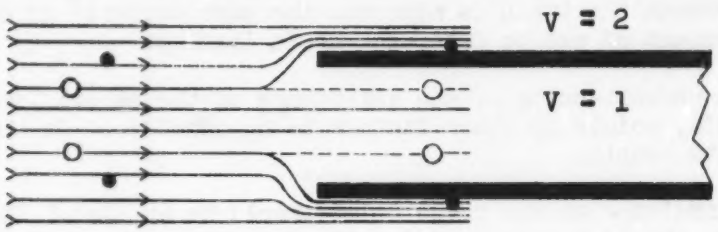

Fig. 1

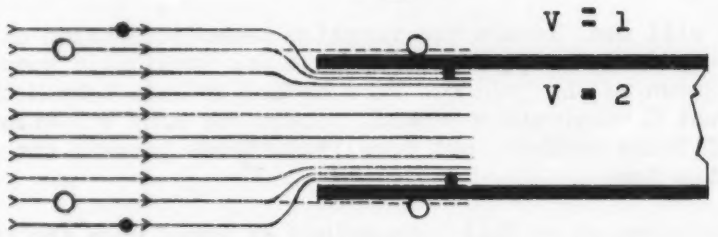

Fig. 2

Figure 1 shows a condition where the velocity of the gas stream inside of the sampling probe is less than the gas velocity within the flue. The air streams, approaching at a higher velocity than the face velocity of the probe inlet, are deflected. Light particles follow these air streams and do not enter the probe. Heavier particles will enter the probe due to inertial effects. This will result in a high concentration of coarse particles being collected.

In Figure 2, the velocity within the probe is higher than that of the main stream. This will cause a convergence of air streams carrying the lighter particles into the probe. These fines would not normally be collected and would thus yield high concentrations of light particles.

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16. Abstracts

Particulate sampling of air/gas streams based on the principles of isokinetic sampling is discussed. Emphasis is placed on the balanced tube (null tube)method and dust concentration method. Gas moving and metering equipment and flow rate meters are also discussed.

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It is, therefore, under most conditions, necessary to maintain the velocity within the probe equal to that of the main stream. (isokinetic conditions) To prove this, tests have been run in order to determine the percentage error when deviating from isokinetic conditions. It was found that where the nozzle velocity ranges from 2/3 of the isokinetic velocity to 2 times that value, the extreme potential error amounts to from 40 to 60 per cent. The dust used in this experiment ranged in size from 5 to 25 μ. It is obvious that the finer the dust, the less sensitive it would be to departures from isokinetic conditions, especially at high probe velocities.

When the velocity within the nozzle is greater than that of the flue, a higher concentration of light particles will be captured. If total weight collected is the only determination made, the error will not be as great as you would expect, since the quantity of gas moving through the train is high and the dust captured is calculated on this amount of gas to determine grain loading.

An understanding of the importance of the stream lines of flow and inertia, points up other factors to be considered in obtaining an accurate sample.

Turbulence at the probe inlet should be avoided. The probe, therefore, should be pointed directly into the stream and the inlet edge beveled.

We will now discuss two sampling techniques based upon the use of the principles just stated; that is, isokinetic sampling. They are known as the Balanced Tube Method or Null Tube Method and the Dust Concentration Method. There are many variations within each of these methods, but time limitations prevent the examination of all but a few.

The Balanced or Null Tube method is based upon the principle, that if the velocity within the sampling nozzle is equal to that outside, the static pressures will be equal.

Two types of null probes that have been developed to sample stacks are illustrated in Figures 4 and 5 of the following paper: Hemeon,W. C. L., Haines,G.F.: The Magnitude of Errors in Stack Dust Sampling,Air Repair,November, 1954, pp. 159-164.

Figure 5 shows the integrated type, where both static taps are in the probe, proper. The probe shown in Figure 4 is less expensive and much simpler to fabricate but obviously more difficult to fit through a sampling hole.

The static tubes are run to opposite sides of an inclined manometer and the sampling velocity adjusted until the manometer reads at a zero point.

This method is not recommended for initial testing but rather may be used for process control after being correlated with results obtained from the Dust Concentration Method.

Large variations in velocity may cause very small variations in static readings. Small variations in static readings may result from slight imperfections in the static holes. It is therefore difficult to obtain a high degree of accuracy using this method.

The procedure, using the Null Tube, is to first make a velocity traverse and fix a point of average velocity. The sampling probe is set at that point and pump adjusted to maintain a zero point reading on the manometer. The time of the run is recorded.

Knowing the cross-sectional area of the probe inlet and the flue, a ratio is set up upon the principle that the ratio of the area of the flue to the area of the probe is equal to the ratio of the weight of a suspensoid passing through the flue to the weight collected in the train (a valid principle if isokinetic conditions are achieved). This leads to the formula for the determination of the suspensoids carried in pounds per hour.

$$\text{pounds per hour} = 0.318 \frac{A \times W_t}{a \times T}$$

Where:

A = Area of the flue in sq. ft.

a = Area of the nozzle in sq. ft.

W_t = Weight of the material caught in grams

T = Duration of the run in hours

Knowing the average velocity in the flue and the area of the flue, the suspensoid concentration may be determined.

The assumption that these ratios exist is true where extreme turbulence is not present at the nozzle and the dispersoid is not concentrated at varying points across the cross section of the flue.

We will now consider a method where the actual velocities are measured within the probe and flue. Adjustments to isokinetic rates are made on the basis of direct velocity readings. This method will be referred to as the Dust Concentration Method.

Other refinements to this method from the Null Tube method include the selection of a nozzle size to provide a good sampling rate and the running of dust sampling traverses. These will be explained more fully, later.

The Sampling Train: A number of factors affect the selection of the type of equipment and its order in the train. A standard all purpose train is non-existent. Each train must be tailored to fit the characteristics of the stack being tested.

For those of you not familiar with exactly what a sampling train may look like, a brief description of a certain type of train will now be presented. The characteristics and methods of selection of the components will follow.

Slide 1

This train is typical of the type that may be used to test an incinerator. The probe is made of Pyrex and is 6 mm in diameter. It is followed by a small glass cyclone, designed to cut out particulate matter at 5 μ and above. There are then three 500 ml impingers set in an ice bath. The first two contain 100 ml of distilled water each. The last is dry and has a thermometer attached to the inside stem. There is a glass "T" off the top of this last impinger. One side leads to a Hg manometer, the other to a Wilson 3-A pump. The pump is provided with a rheostat to control the sampling rate. Following the pump is a Sprague Meter, provided with its own Hg manometer and thermometer. The last component is a flow meter calibrated to read CFM. The inclined gauge to the extreme left is used with the "S" tube in the foreground to make the velocity traverse.

We will now start at the probe and examine the various components of the train.

Probes may be obtained commercially such as the Western Precipation Model. These are supplied in various sizes and lengths with interchangeable nozzles.

Pyrex probes, such as the one shown may be easily fabricated. These may be provided with ball and socket joints to accept extension lengths. They are stocked usually in sizes from 5 to 12 or 13 mm in diameter.

For high temperature rugged work, Vycor glass tubes are excellent. These require special techniques for bending.

Glass probes have several advantages.

1. They will resist corrosive condensates which may form inside the probe.

2. They are relatively less expensive than the metal probes.

3. They can be cleaned easily and any holdup in the tube can be seen.

Metal probes are best for heavy rugged work and extremely high temperatures.

Slide 2

Following the probe, it may be advisable to have an auxiliary collecting device (or prefilter). This of course depends upon the estimated grain loading in the stack. If the grain loading is heavy, these auxiliary devices will prevent the more efficient filter, following, from getting clogged. The prefilter shown on the slide is a glass cyclone; not too efficient for small particles, but useful in reducing the dispersoid concentration going to the impinger and filter and cutting out large particles which may plug up the impinger orifice. Almost any type of coarse filtering material in a suitable holder will make a good prefilter. This small cyclone is excellent. It can be cleaned out fairly well and may be used to classify the material collected. A glass fiber sock in a proper holder may be used. Impingers have been found to be effective. In an ice bath, they can collect condensibles or when provided with a solvent or other reactant, they can remove oil or chemical mists.

Condensation of moisture in dry collection prefilters or after-filters should be avoided. Liquid droplets may cause agglomeration of particles and clogging of filter pores. To prevent this, when sampling hot gases having a high dew point, the prefilter should be heated. This may be accomplished using laboratory heating tape, by enclosing the unit in an asbestos board box provided with a resistance heating element or by keeping the filter and holder within the hot stack. Another method is to bleed metered hot air into the system, before the filter.

An impinger train in an ice bath will lower the dew point of the gas-- and as was shown in the slide, the last impinger (Slide 2), which was dry, would remove liquid droplets. This last impinger was provided with a thermometer and a manometer. The gas in this impinger would be saturated and knowing the temperature and pressure of the gas, its moisture content may be found.

The afterfilter or efficient separator is chosen to give the best collection of the dispersoid being sampled considering the conditions of the media in which it is dispersed.

The Whatman paper thimble is used quite often. These extracted thimbles are 43 X 123 mm and are accurate, inexpensive and convenient to use. When kept dry, they will operate well at temperatures up to 350°F. and 4 inches Hg suction. Heavy dust loading will clog the filter.

It was for the above reasons that the paper filter was placed after the impingers in the incinerator train, as shown in Slide 1. The impingers lowered the dew point temperature of the gas and reduced the grain loading in the stream going to the paper filter. Condensation may also be controlled by heating the holder, as already mentioned.

The paper filter may be held in a standard holder manufactured by the Western Precipitation Corporation. A holder may be easily made out of pyrex glass as shown in Figure 3. This holder is inexpensive and has the advantage that the thimble may be examined for breaks during the sampling.

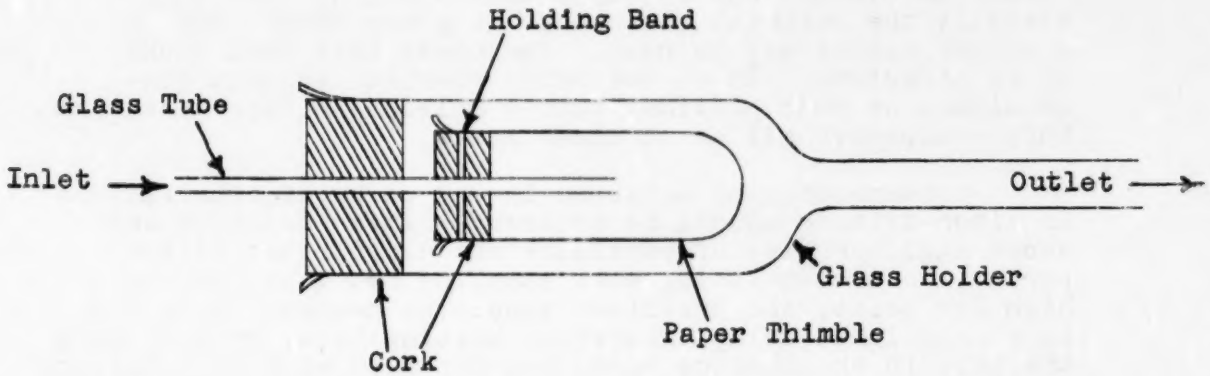

Fig. 3

In the lab, the paper filter will be used with a glass wool sock as a prefilter.

Errors sometimes result from unremoved dust "holdup" in long probes. It is evident that a short probe is desirable. This would mean, in some instances, the filter would have to be placed within the stack. A filter material and housing have been developed which can withstand all the high temperatures that may be reasonably expected. This is the alundum filter. These thimbles are available in medium or coarse porosities and two common sizes, 34 X 100 mm and 45 X 127 mm. They are resistant to acid gas, can be subjected to temperatures of over 1000°F. and can be weighed with a high degree of accuracy.

The alundum filter does have some drawbacks. It is brittle and has a high resistance to air flow. This resistance may be reduced by placing what is in essence a prefilter within the thimble. Pyrex glass wool may be used to line the thimble. The glass wool aides in filtering and prevents pile-up on the thimble wall. It also allows the thimble to be used many times. By placing the alundum thimble within the hot stack, the heating of the holder by

the hot stack gases will prevent condensation. A heating box may also be used (Slide 3 - Close-up of holder and heater box) when a long probe is used.

The impinger method for collecting the sample is employed whenever the dust encountered is wettable. The 500 ml size is usually used. The impinger can be used effectively as a bubbler when testing in acid or basic atmospheres--100 ml of a 5 per cent solution of NaOH or HCl in an impinger is used to test an acid or basic atmosphere respectively. The analysis is performed by filtration. A 1 per cent solution of $NaHSO_3$ in an impinger can be used as a test for aldehydes. The analysis is performed by idiometric titration. Important: An impinger should be avoided when there is a chance of chemical-reaction taking place between components of the gas stream being sampled. The impinger provides excellent conditions in which reactions may take place. These reactions may not take place in the plume itself and would thus yield erroneous results.

A description of the standard impinger is given in Public Health Bulletin #217: U.S. Public Health Reports. Volume 52, No. 33, August 13, 1937.

The collection efficiency of the impinger is greatly effected by the rate of sampling. When attempting to capture dust using an impinger, a rate of from 0.5 to 1.0 CFM has been found to be efficient. Most of the other filters used commonly in a train operate best within this range, so no difficulty would be encountered in using the impinger in series.

It is for effective dust separation that trains are operated at approximately 1.0 CFM and it is for this reason that various nozzle sizes must be used to obtain isokinetic sampling, that is, the velocity at the nozzle inlet equal to the stack velocity. Adjusting the rate at the pump beyond the recommended rates, usually from 0.5 to 1.5 CFM will affect filter collection efficiency. The rates of course do not apply to specially designed high volume equipment operating at about 5 to 10 CFM and now mostly in the experimental stage.

Filters of finely woven glass cloth are excellent prefilters with coarse dust. They are not efficient for the collection of fumes.

The filter holder for the glass cloth sock is similar to the Whatman thimble holder, only larger. The sock will operate effectively up to a pressure drop of 6 inches Hg and temperatures as high as 750°F. The pores of the filter are easily clogged by moisture. These filters may be used several times if cleaned and weighed properly. The sock shown is supplied by Western Precipitation Corporation,

but several variations have been fabricated for special use. Homemade holders, similar to the glass Whatman thimble holder may be easily fabricated.

The electrofilter is a small, high efficiency Cottrell Precipitator through which the gas being sampled is drawn at a low velocity. An aluminum cylinder 1 1/2" I.D. is used as the collecting electrode. A small wire running through an insulated bushing and down the middle of the cyclinder is used as the discharge electrode. A rectified voltage of approximately 20,000 volts is provided by a power pack using a vacuum tube rectifier and condenser system. The rate of gas flow through the unit is between 1/2 and 1 CFM. The lower the gas rate the more efficient will be the collection. The electrofilter has some limitations. It should not be used when testing very corrosive gases or in a combustible atmosphere. It is subject to all the factors affecting collection in a standard electrostatic precipitator; humidity conditions of the gas, moisture content of the dust and grain loading. It is important to maintain the precipitator temperature above the dew point of the sampled gas to prevent moisture condensation in the precipitator.

A precipitator has been constructed by the Los Angeles County Air Pollution Control District. It consists of 12 test tubes - 18" long and 2" in diameter. These tubes have a side neck tube sealed on 3" above the base of the tube for drawing the air sample through. A space below the side tube serves as a reservoir for any dust particles which may drop off the side wall after collection . Stainless steel discharge electrodes are suspended from a bakelite sheet 3" above the top of the tubes. These electrodes are so constructed that they may be individually centered in the tubes by means of lock nuts. The ground electrodes are made of tin foil and placed outside of the tubes by means of lock nuts. The ground electrodes are made of tin foil and placed outside of the tubes to prevent metal contamination of the collected dust. Power is provided by a 20,000 volt power pack and a rate of 2 CFM per tube has been found to give efficient collection. The tubes are placed in parallel; therefore, a total rate of 24 CFM or 1440 CFM may be sampled.

I might interject, before going into chemical absorption filters, that the U. S. Bureau of Mines has performed some stack sampling operations using a millipore or membrane filter. This filter is used at present for microscopic examination of dust only.

Chemical Absorption Filters: As has been mentioned, adsorption may be carried out using impingers. For example, in testing for NH_3 - three impingers may be used. The first two containing at 5 per cent solution of HCl. After

sampling, the solutions in the impingers are combined. An excess quantity of NaOH is added to this solution and the NH_3 distilled into a measured volume of standard acid. The excess acid is back titrated with a standard base.

Oil mists and vapors may be conveniently absorbed in CCl_4. The apparatus consists of five impingers in an ice bath and a paper thimble at the end of the line. The first impinger is run dry in order to collect any water vapor or heavy oil drops. The fifth impinger is also run dry in order to insure that the thimble will be maintained dry. CCl_4 is placed in the second, third and fourth impingers. If the CCl_4 is evaporated during the run, more must be added. At the completion of the test, the solutions are carefully evaporated and the oil weighed. The vapor volume of the CCl_4 evaporated during the run must be subtracted from the meter reading. Another method for determining oil mist is to use a piece of apparatus known as a tar camera, fully described in Bulletin WP-50 of the Western Precipitation Corporation.

Many other determinations dependent upon absorbing some component of the sampled gas stream in a reactant with the use of an impinger train are possible.

The use of a 1 per cent sodium bisulfite solution in testing for aldehydes has been mentioned. Bubbling through a 1 per cent phenylhydrazine hydrochloride solution is a good test for formaldehyde. An idiometric determination (magenta color) is made with this test. A phenoldisulfonic acid solution may be used in the determination of oxides of nitrogen.

A Woulff bottle may be used in place of the impinger when high sampling rates are desired. The bottle may be packed with glass beads or rings.

Adsorption may be carried out using preweighed cartridges of activated carbon, silica gel or ascarite. Adsorption presents a problem of nonselective separation due to mechanical holdup in the cartridge.

Absorbing solutions in evacuated bottles may be used for obtaining grab samples. These samples may be taken at the same time that the sampling train is operating.

Slide 4

Shown on this slide is a 250 ml evacuated mine safety bulb. This bulb does not usually contain an absorbing solution and may be used for collecting samples for hydrocarbon analysis by IR or MS. Also shown is a two-liter evacuated bottle. Various types of absorbing solutions may be placed in this bottle for the determination of stack constituents. For example, for aldehydes, the

bottle may contain a one per cent sodium bisulfite solution;for oxides of nitrogen, a mixture of H_2O_2 and H_2SO_4 (analysis by the phenoldisulfonic acid method.) The two-liter evacuated bottle may also be used for dry collection for IR and MS analyses. A 500 ml evacuated bottle with a rubber cork that accepts a hypodermic needle is shown in the background. An absorbing solution is placed within the bottle and a hypodermic needle is used to draw the sample into the container. Capillaries may be used as entrance ports on evacuated bottles to increase the time of sampling. Also shown on the slide is a bottle used for taking an integrated gas sample for Orsat analysis. This sample is collected by withdrawing gases continuously from the stack at a constant rate by a liquid displacement method. The five-liter bottle is filled with a saturated solution of sodium sulfate acidified with sulfuric acid. As the solution is siphoned to another container, the gases enter the bottle and are later run on an Orsat apparatus.

Glass Paper Felted Filter

The Engineering Section of the Community Air Pollution Program is developing a high volume stack sampler. The prefilter in the unit is a small high efficiency cyclone. The second filter unit in the train will be referred to as an absolute filter. This fiberglas web filter is a high efficiency filter and is effective in collecting metal fumes. The filter is made by the Mine Safety Appliance Company (No. 1106). It is flash-fired to remove any bonding materials--thus making the filter non-hygroscopic.

Gas Moving and Metering Equipment

Slide 5

This slide shows the aft end of the train. A Wilson 3-A is shown, followed by a Sprague meter and then a flow meter. The Wilson pump shown is supplied with a rheostat to adjust the sampling rate. The pump operates very well between 1/2 and 1 1/2 CFM, and will handle any pressures usually encountered in a train. A rheostat provides fine adjustment of the sampling rate. A pinch clamp, placed on the rubber hose before the pump, may also be used. Another method of adjusting the sampling rate would be to use an air bleed-in valve before the pump. This bleed-in valve could only be used if the pump were at the end of the line, since the air that is being metered should be stack gases only. It may be possible to place a meter on the bleed-in line but this would be cumbersome.

The gas rate control for the high volume sampler is achieved by a by-pass valve. This will be more fully described in the lab. The pump used in the lab and at

the industrial source (with the thimble set up) is a Gast rotary pump. It will operate well at 2 1/2 CFM at 20-25 inches mercury Hg, depending upon the length of the run. Ejectors may also be used to supply suction for the train. The use of water, steam or air ejectors is recommended when sampling in locations, such as refineries, where the use of electrical equipment is not desirable. A water ejector is described in Bulletin WP-50. Water, steam and compressed air-driven pumps may also be used.

It is necessary to have an integrated meter on the line to record the total gas flow through the train.

Two types of integrating meters are in general use in stack sampling trains.

One is the wet test meter. Obviously, if the gas being sampled is soluble in water, this meter should not be used. The leveling of the meter and setting of the water level are critical. The meter is also quite heavy and bulky.

The Sprague meter shown in Slide 5 is the one that is generally used in stack testing. This is a dry type meter. The integrated mechanism is activated by calibrated leather billows being expanded alternately. It is lighter than the wet test meter and does not require leveling. This meter is provided with a manometer and a thermometer on its outlet side. You will remember, a dry impinger was used in the train. The gases going through this dry impinger were saturated while passing through the integrating meter. Therefore, a knowledge of the temperature and pressure at the meter will be needed to correlate the flow recorded with the rate of gas intake at the sampling nozzle. The volume of air passing through the nozzle is not necessarily the same as the volume measured at the meter. This may be due to condensation (in the impinger trains) or temperature changes in the line. The change in volume of the gas passing through the train and compensations to be made in determining the meter reading will be discussed more fully by the next lecturer.

If the pressure at the meter were known in advance calculations to determine what meter reading would yield isokinetic sampling rates would be much simpler. The meter is therefore usually placed at or near the end of the line, after the pump, so that the pressure on the outlet side of the meter is small and relatively constant.

Flow Rate Meters

The integrating meter described above records the total volume throughput at meter conditions. With the

use of a stop watch it is possible to measure a rate using the integrating meter. Let us consider an actual field sampling procedure. The train is set up and the probe is in the stack. The volume rate (at the probe) to be pulled through the sampling system in order to give isokinetic conditions has been determined. Knowing the temperature and humidity of the gas being sampled and the pressure at the meter, the rate is corrected to obtain the actual meter reading. We now know the rate that should be read at the meter. The pump is started, an initial reading is taken at the meter and another reading after a given elapsed time (two to three minutes). Assume the sampling rate is found to be too high. The screw clamp or rheostat is adjusted to lower the sampling rate. The rate at the meter is checked again and found correct. As the sampling continues, the material being collected builds up on the filter and the resistance in the train increases. The sampling rate drops off. Therefore, constant surveillance must be maintained at the meter and adjustments made. There was a lag in time between the change in rate occuring in the train and the compensating adjustment when rates are read off the integrating meter. In certain cases, of varying stack velocities, a Pitot tube is maintained in the duct throughout the run and adjustments in the train sampling rate have to be made quite often. It is obvious from this discussion that it would be advantageous to have a flow meter in the line reading rates (CFM), instantaneously. There are two types of meters generally employed in stack sampling operations. The flow meters are placed on the clean air side of the filtering unit--usually at the tail end of the train. One is a rotameter, which must be used in a vertical position.

An orifice meter is used most often in the field.

A constriction in the line or flue maintains a differential pressure across the U tube tap holes. This differential pressure can be related to give readings in CFM.

We have now discussed the train make-up, starting with the probe and ending with the flow rate meter. It would be well to review some of the factors affecting train make-up, as a conclusion to this talk.

It should be borne in mind that laboratory work must follow any sampling operation. The simplifying of laboratory analysis is important. Therefore, a minimum number of filtering units is desirable. If the grain loading is light and no condensation is expected at the filter, one filtering unit may be sufficient. If classification of the material to be sampled is desired, pick a prefilter or number of prefilters that will cut out the size fractions of interest. This, again, will simplify laboratory particle classification techniques.

Consider the carrying gas when choosing the filter. A paper thimble may effectively collect the size particle matter being sampled, but if the carrying stream is extremely corrosive or at a high temperature, an alundum thimble may have to be substituted.

The use of the impinger as a condenser has been discussed. Three factors to be considered in using the impinger are:

1. The sampling rate is limited to around one CFM if the impinger is to take part in collection.

2. A prefilter is required to cut out large particles which may plug the impinger orifices. Most inertial prefilters in standard use have efficient rates of collection corresponding with the recommeded rate of the impinger.

3. Consideration must be given to the solubility of the material being collected for a reaction that might take place in the impinger solution.

NOTE: An impinger offers ideal conditions for a reaction to take place.

A standard coil tube condenser may be used in the place of an impinger. The coil tube condenser, of course, cannot be used to collect a sample and must be placed after an efficient filter.

Much leeway is given in the choosing of metering and pumping devices. The equipment usually employed has been described, but it must be remembered that stack sampling techniques are fairly new and there are many other efficient devices on the market that have not been tried.

There are basic principles which will determine the choosing of components of a train and, with experimence, an appreciation will be gained for the limitations and characteristics of these components. Again, it should be pointed out that there is no such thing as a standard train that may be used for all stack sampling operations.

Some of the basic concepts of collection to be observed are:

1. The sampling should be at a rate most efficient for collection. If a number of sampling components are in series, they must all operate at maximum efficiency at approximately the same volume rate.

2. Each filtering component should be operated at temperatures, pressures,and humidity conditions recommended for that particular component.

3. The temperature and pressure conditions of the effluent being sampled must be known at the metering points.